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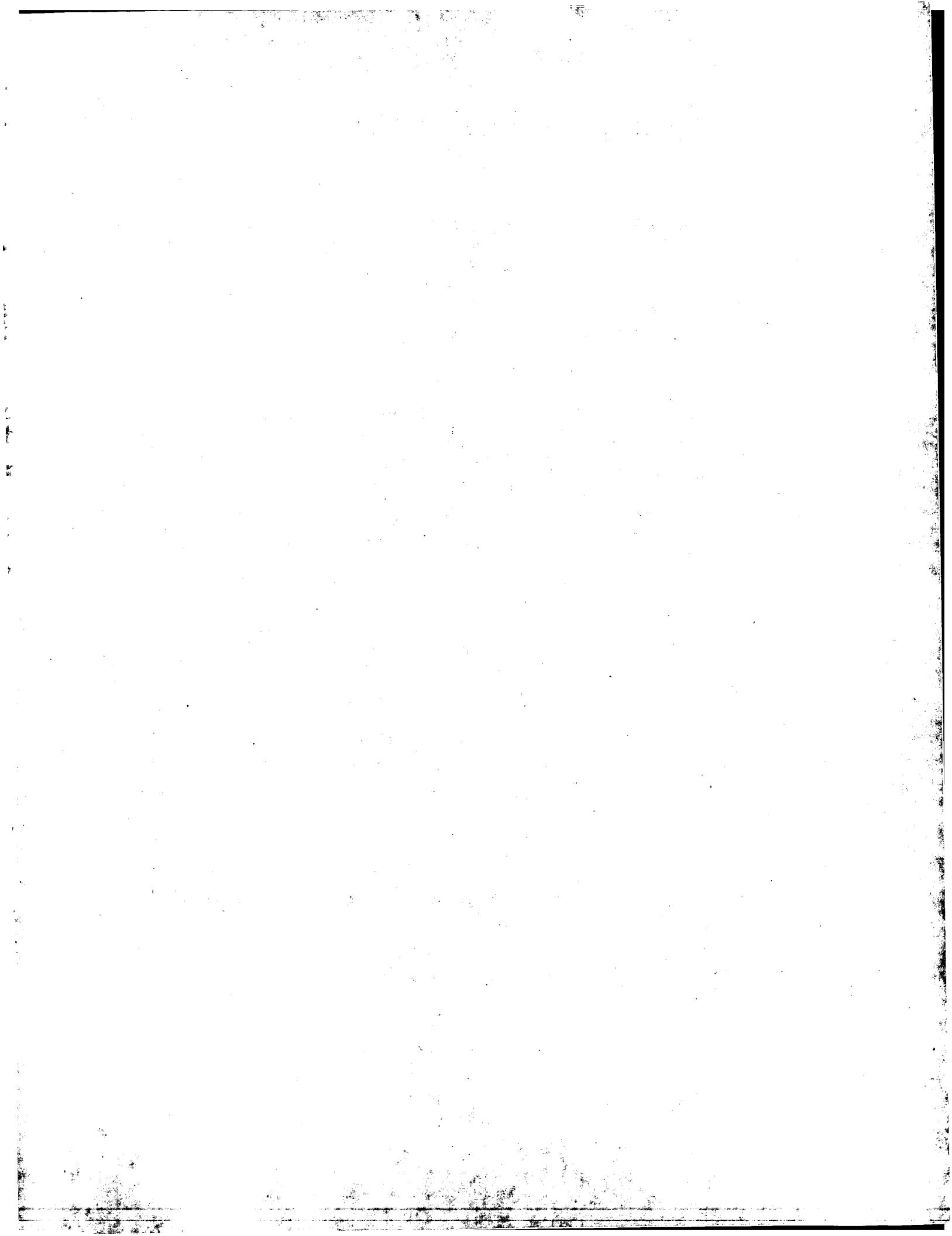
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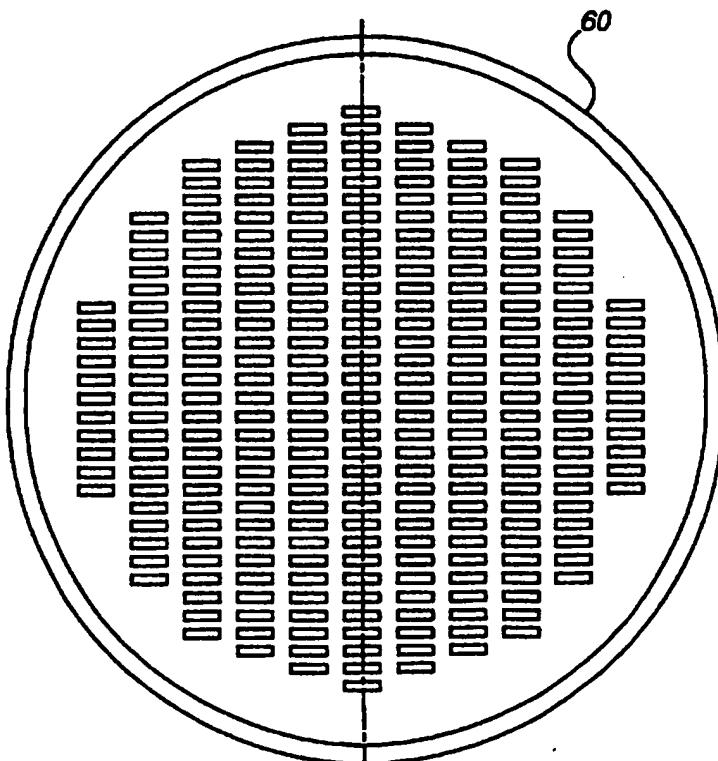


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<p>(21) International Application Number: PCT/US96/00884</p> <p>(22) International Filing Date: 24 January 1996 (24.01.96)</p> <p>(30) Priority Data: 08/379,877 27 January 1995 (27.01.95) US</p> <p>(71) Applicant: MINE SAFETY APPLIANCES COMPANY [US/US]; RIDC Industrial Park, 121 Gamma Drive, Pittsburgh, PA 15238 (US).</p> <p>(72) Inventor: FRUND, Zane, N.; 212 Saint Andrews Court, Cranberry Township, PA 16066 (US).</p> <p>(74) Agent: BARTONY, Henry, E.; Bartony Hare & Edson, The Bank Tower, Suite 801, 307 Fourth Avenue, Pittsburgh, PA 15222 (US).</p>		<p>(81) Designated States: AM, AT, AU, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IS, JP, KE, KG, KP, KR, KZ, LK, LR, LT, LU, LV, MD, MG, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TT, UA, UG, UZ, VN, ARIPO patent (KE, LS, MW, SD, SZ, UG), European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).</p> <p>Published <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p>	
<p>(54) Title: RESPIRATOR FILTER SYSTEM</p> <p>(57) Abstract</p> <p>The present invention provides a respirator filter system suitable for filtering toxic agents, including organic vapors, acid gases, formaldehyde, ammonia, and methylamine as well as pesticides, CS and CN tear gases, dusts, mists, fumes and radionuclides from environmental air to be respired by a user of the filter system. In general, the filter system of the present invention includes a cartridge (5) containing a layer (20) of an unimpregnated activated carbon, a layer (30) of an activated carbon impregnated with sulfate, molybdenum and at least one impregnate selected from the group consisting of copper and zinc, and a HEPA filter. Cartridges (5) containing these adsorbents or adsorbents similar to them (in carbon tetrachloride activity and chemistry) have been discovered to be uniquely capable of filtering a broad range of toxic agents.</p> 			

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TITLE

RESPIRATOR FILTER SYSTEM

Field of the Invention

The present invention relates to respirator filter systems and, in particular, to respirator cartridges suitable for use in environments containing numerous types of toxic agents.

Background of the Invention

Air-purifying respirators equipped with filter systems such as chemical cartridges have been used for decades to protect the respiratory system of individuals from noxious gases, vapors, fumes and particulates. Typically, respirator manufacturers in the United States produce chemical cartridges approved by the National Institute of Occupational Safety and Health (NIOSH) for either a single type of toxic agent (for example, pesticide, organic vapor, acid gas or formaldehyde) or for multiple types of toxic agents (for example, organic vapors and acid gases, ammonia and methylamine, or acid gases and formaldehyde). Similarly, respirator manufacturers around the world produce chemical cartridges for approval by appropriate regional or national regulatory agencies such as NIOSH or CEN.

Generally, these cartridges contain adsorbents such as activated carbon (for example, coal-, peat- or coconut-based activated carbon) for the physical adsorption of toxic agents such as organic vapors, and/or various types of impregnated carbons (for example, sodium hydroxide, potassium iodide and zinc chloride impregnated carbon) for the

chemisorption of toxic agents such as acid gases, formaldehyde, ammonia and methylamine.

Several respirator manufacturers, in an attempt to broaden the range of toxic agents against which respirator 5 filter systems are effective, produce "dual fill" cartridges comprising a layer of an activated carbon and a layer of impregnated activated carbon. For example, Mine Safety Appliances Company of Pittsburgh, Pennsylvania produces a GMF filter system suitable for use in atmospheres containing 10 organic vapors, acid gases and formaldehyde.

However, no single type of air-purifying respirator filter system is currently available which is capable of filtering a broad range of toxic agents, including organic vapors, acid gases, formaldehyde, ammonia, methylamine and 15 pesticides, well enough to meet NIOSH requirements. A cartridge which adsorbs all of the aforementioned toxic agents would be very valuable to individuals potentially exposed to a myriad of toxic compounds as, for example, in the agricultural industry.

20

Summary of the Invention

Accordingly, the present invention provides a filter system suitable for filtering a broad range of toxic agents, including, for example, organic vapors, acid gases (for example, chlorine, chlorine dioxide, hydrogen chloride, 25 hydrogen sulfate and sulfur dioxide), formaldehyde, ammonia, methylamine and pesticides from environmental air to be respired by a user of the filter system. The air-purifying filter systems of the present invention comprise containers or cartridges containing a novel mixture, ratio, and arrangement 30 of chemical adsorbents.

In general, the cartridges of the present invention contain a layer of an unimpregnated activated carbon preferably having a carbon tetrachloride activity of at least 85 and a layer of an activated carbon impregnated with a 5 sulfate compound, a molybdenum compound and at least one compound selected from the group consisting of a copper compound and a zinc compound. Preferably, the impregnated carbon should include by weight: up to 10% sulfate and up to 10% molybdenum and at least one impregnate selected from the 10 group of copper and zinc, wherein the copper is present in an amount from 0.0 to about 20% and the zinc is present in an amount from about 0.0 to 20%. Cartridges containing such adsorbents or adsorbents similar to them in carbon tetrachloride activity and chemistry have been discovered to 15 be uniquely capable of filtering a wide range of toxic agents much broader than that of any existing cartridge.

Preferably, in the respirator cartridge filter systems of the present invention, there is at least 20 cc of activated carbon in the layer of unimpregnated activated 20 carbon and at least 80 cc of impregnated carbon in the layer of impregnated activated carbon. More preferably, there is at least 26 cc of the activated carbon and at least 87 cc of the impregnated carbon present in the activated carbon layer and the impregnated carbon layer, respectively. Preferably, the 25 unimpregnated carbon is placed downstream from the impregnated carbon. The respirator cartridges of the present invention meet all the NIOSH requirements for organic vapors, acid gases, formaldehyde, ammonia and methylamine. Moreover, when the cartridges of the present invention are equipped with a 30 high-efficiency particulate (HEPA) filter, the present cartridges also meet the NIOSH requirements for pesticides, CS and CN tear gases, dusts, fumes, radionuclides, lacquers and enamel mists. No existing respirator cartridge meets all of these NIOSH requirements.

As set forth in Title 30, Part 11 of the United States Code of Federal Regulations and as used herein, the term "vapor" means the gaseous state of a substance that is solid or liquid at ordinary temperature and pressure. The 5 term "gas" means an aeriform fluid which is in a gaseous state at ordinary temperature and pressure. The term "facepiece" refers to a respirator component designed to provide a gas-tight or dust-tight fit with the face and may include headbands, valves, and connections for canisters, cartridges, 10 filters, or a respirable gas source. The term "dust" means a solid mechanically produced particle with a size ranging from submicroscopic to macroscopic. The term "fume" means a solid condensation particle, generally less than 1 micrometer in diameter. The term "mist" means a liquid condensation 15 particle with a size ranging from submicroscopic to macroscopic.

The term "pesticide" means (1) any substance or mixture of substances (including solvents and impurities) intended to prevent, destroy, repel, or mitigate any insect, 20 rodent, nematode, fungus, weed, or other form of plant or animal life or virus, and (2) any substance or mixture of substances (including solvents and impurities) intended for use as a plant regulator, defoliant, or desiccant, as defined in the Federal Insecticide, Fungicide, and Rodenticide Act of 25 1947, as amended (7 U.S.C. §§135-135k), excluding fumigants which are applied as gases or vapors or in a solid or liquid form as pellets or poured liquids for subsequent release as gases or vapors.

The term "radionuclide" means an atom identified by 30 the constitution of its nucleus (specified by the number of protons Z, number of neutrons N, and energy or, alternatively, by the atomic number, mass number $A=(N+Z)$, and atomic mass) which exists for a measurable amount of time; decays or

disintegrate spontaneously, emits radiation, and result in the formation of new nuclides.

Brief Description of the Drawings

Figure 1 illustrates in cross-section an embodiment 5 of a respirator cartridge of the present invention.

Figure 1A is a top plan view of a lid for the respirator cartridge of Figure 1.

Figure 2 illustrates in cross-section an embodiment 10 a respirator cartridge of the present invention including a high-efficiency particulate filter.

Detailed Description of the Present Invention

The respirator filter systems of the present invention can vary widely in shape (for example, round, rectangular, oval, etc.) and size (including, fill volume), 15 depending upon their materials of construction and the performance criteria (for example, the desired service time and the desired breathing resistance, and the amount of dust, mist and fumes to be removed from an airstream). However, the geometry and size of the respirator cartridge should not 20 obstruct the wearer's eyesight when mounted on the respirator facepiece. In general, it is desirable to minimize the volume of carbon used in the filter system while maximizing the gas life of the filter system.

25 Figures 1 and 2 illustrate two preferred embodiments of the present respirator filter system. As illustrated, cartridge 5 comprises a round cartridge body 10. Examples of suitable cartridge bodies 10 include the Advantage® cartridges

or Comfo® cartridges available from Mine Safety Appliances Company of Pittsburgh, Pennsylvania. See Mine Safety Appliances Company Data Sheet Nos. 10-02-03, 10-02-04 and 10-02-05.

5 Pica USA G215D and Calgon Carbon URC are examples of an unimpregnated activated carbon and an impregnated activated carbon, respectively, suitable for use in the present invention. The physical and chemical characteristics of a commercially available Pica USA G215D carbon and a
10 commercially available Calgon Carbon URC carbon used in the present studies are presented in Table 1.

Calgon Carbon URC is the subject of Patent Cooperation Treaty International Application No. PCT/US92/10216, the disclosure of which is incorporated herein by reference. Adsorbents having a carbon tetrachloride activity and chemistry similar to these adsorbents should also be suitable for use in the respirator cartridges of the present invention.

TABLE 1

CHARACTERISTICS OF UNIMPREGNATED AND IMPREGNATED CARBONS

	Unimpregnated Carbon	Impregnated Carbon
Base Carbon	coconut	Coal
Carbon Tetrachloride Activity	85 +	60 +
Mesh Size	12 x 20	12 x 30
Apparent Density gm/cc	0.42	0.66
Impregnates	none	12% copper 7% ammonium sulfate 6 % ammonium dimolybdate
Supplier	Pica USA G215D	Calgon Carbon URC

Calgon Carbon URC impregnated activated carbon is known to be somewhat effective in the removal of organic vapors, acid gases, formaldehyde, ammonia and methylamine. Such impregnated carbon may comprise by weight: up to 10% sulfate and up to 10% molybdenum and at least one impregnate selected from the group of copper and zinc, wherein the copper is present in an amount from 0.0 to about 20% and the zinc is present in an amount from about 0.0 to 20%.

The Pica USA G215D and Calgon Carbon URC adsorbents were selected over other adsorbents for use in the present invention because of several advantages, including (i) a large surface area and micropore volume, and thus a high absorptive capacity for organic compounds, (ii) the ability after impregnation as described above to chemically adsorb a wide range of toxic agents, and (iii) an acceptable breathing resistance (pressure drop) when placed in a cartridge.

To meet current NIOSH gas life requirements for organic vapors, acid gases, formaldehyde, ammonia, methylamine, cartridge 5 must contain a layer 20 of at least approximately 20 cc of an activated carbon having a carbon 5 tetrachloride activity of at least 85, and a layer 30 of at least approximately 80 cc of a carbon impregnated with a sulfate compound, a molybdenum compound and at least one compound selected from a group consisting of a copper compound and a zinc compound. When present in these amounts, the layer 10 of unimpregnated carbon is preferably placed downstream of the layer of impregnated carbon to meet NIOSH requirements. To contain the adsorbent bed, cotton or synthetic filter pads 40 are preferably placed underneath and on top of the adsorbent bed.

15 If protection against dusts, fumes, radionuclides, lacquers and enamel mists is desired or required, a high efficiency particulate (HEPA) filter 100 (for example, a pleated, electrostatic or conical-shaped filter) can be attached to the top of cartridge 5.

20 Further, present NIOSH requirements require a HEPA filter for NIOSH approval against both pesticides and tear gases. Satisfaction of the NIOSH requirements for both pesticides and tear gases in a single filter system thus require both a carbon bed as describe above and a HEPA filter.

25 Examples of a suitable HEPA filters for use in the present invention include (i) MSA HEPA filters, which are fabricated from a non-woven blend of microglass and synthetic fibers, and (ii) electrostatic HEPA filter media, such as the 30 Web Dynamics WD2210 synthetic filter media. In general, HEPA filters suitable for use in the present invention are preferably at least 99.97 percent efficient against 0.3 micron dioctyl phthalate (DOP) aerosol particles.

To construct cartridge 5, at least one filter pad 40 is preferably placed in the bottom of cartridge 5. More preferably, at least two filter pads 40 are placed in the bottom of cartridge 5. At least approximately 20 cc of the 5 unimpregnated carbon 20 followed by at least approximately 80 cc of the impregnated carbon 30 are then measured and preferably "sifter or vibratory" filled (as known in the art) on top of bottom filter pad(s) 40. At least one filter pad 40 is then placed on top of the impregnated carbon bed. Suitable 10 filter pads are available, for example, from American Felt and Fiber. Finally, a porous (for example, louvered) lid 60 is placed on the top of cartridge 5 and sealed thereon. This procedure compacts the chemical bed, and maintains a suitable bed pressure (that is, a pressure suitable to prevent movement 15 of the carbon bed within cartridge 5).

In cases in which cartridge 5 must also protect against pesticides, tear gases, dusts, fumes, radionuclides, lacquers or enamel mists, a non-woven, high efficiency 20 particulate filter 100 is preferably attached to the top of cartridge 5 as shown in Figure 2.

PERFORMANCE

The present cartridges were tested against the regiment and requirements established by NIOSH as set forth in 25 Tables 2, 3 and 4. Approval by NIOSH is a necessary prerequisite for a respirator cartridge to be commercially viable in the United States. The NIOSH requirements and testing conditions are detailed in Title 30, Part 11 of the Code of Federal Regulations, the disclosure of which is 30 incorporate herein by reference.

The "gas life" of a cartridge is defined as the time that a cartridge is challenged with a specified contaminant until the contaminant "breaks through" the cartridge and is detected in the effluent airstream at a predetermined 5 "breakthrough concentration". The breakthrough concentration of the contaminant is usually its threshold limit value (TLV). The "breathing resistance" is defined as the pressure drop of air flowing through the cartridge at a fixed flow rate. The "challenge agent penetration" is defined as the mass or 10 percentage of challenge particles which are not filtered by the cartridge.

TABLE 2

NIOSH GAS LIFE TESTING CONDITIONS AND REQUIREMENTS

15

Test Agent	Challenge Concentration (ppm)	Breakthrough Concentration (ppm)	Minimum Required Gas Life (min.)
CCl ₄	1000	5	50
SO ₂	500	5	15
HCl	500	5	25
Cl ₂	500	5	17.5
ClO ₂	500	0.1	30
H ₂ S	1000	10	30
HCN	500	5	30
CH ₂ O	100	1	50
NH ₃	1000	50	25
CH ₃ NH ₂	1000	10	12.5
HF	70	3	30
CS TEAR GAS	3	0.05	480
CN TEAR GAS	16	0.05	480

TABLE 3

**NIOSH HIGH EFFICIENCY FILTER TESTING
CONDITIONS AND REQUIREMENTS**

Test Agent	Challenge Conditions	Maximum Allowable Challenge Agent Penetration
Diocetyl phthalate (DOP) penetration	100 $\mu\text{g}/\text{l}$ of 0.3 μ diameter DOP particles at 42.5 lpm for 10 seconds	0.03%
Silica Dust Penetration	60 mg/m^3 of 0.4-0.6 μ diameter silica particles at 16 lpm for 90 minutes	1.5 mg
Silica Mist Penetration	25 mg/m^3 of 0.4-0.6 μ diameter silica particles at 16 lpm for 312 minutes	2.5 mg
Lead Fumes Penetration	20 mg/m^3 of lead particles at 16 lpm for 312 minutes	1.5 mg

TABLE 4

NIOSH BREATHING RESISTANCE TEST CONDITIONS AND REQUIREMENTS

	Chemical Cartridge	Chemical Cartridge with HEPA
Maximum Breathing Resistance at 42.5 lpm (mm H ₂ O)	40	50

The performance of cartridges of the present invention in several experiments is presented in Tables 5 and 5 6. In the experiments represented in Tables 5 and 6, all cartridges were tested at an air flow rate, temperature and relative humidity of 32 lpm, 25°C and 50%RH, respectively. The breathing resistance studies set forth in Table 6 were performed using MSA Comfo II and Advantage 100 cartridges both 10 with and without MSA HEPA filters.

The MSA Comfo II and Advantage 100 cartridges have a fill volume in the range of approximately 115 to 120 cc. In the studies set forth in Tables 5 and 6, the Comfo II cartridges were filled with approximately 26 cc of the 15 unimpregnated activated carbon and approximately 88 to 90 cc of the impregnated activated carbon. The Advantage 100 cartridges were filled with approximately 26 cc of the unimpregnated activated carbon and approximately 92 cc of the impregnated activated carbon.

20 All of the cartridges tested met the gas life and breathing resistance requirements set forth by NIOSH. Moreover, all the cartridges tested exhibited similar gas life

performances. When a non-woven, HEPA filter 100 was incorporated into cartridge 5, as shown in Figure 2, cartridge 5 also met the NIOSH requirements for pesticides, ortho-Chlorobenzylidene (CS) and alpha-Chloroacetophenone (CN) 5 tear gases, dusts, mists, fumes and radionuclides. Furthermore, when tested at a phosphine (PH₃) challenge concentration of 1500 ppm (at 32 lpm, 50% RH and 25°C), all the cartridges exhibited a gas life of 30+ minutes with a breakthrough concentration of 0.3 ppm.

TABLE 5

**PERFORMANCE OF THE PRESENT CARTRIDGES
AGAINST NIOSH TESTING CONDITIONS**

Test Agent	Service Time Minutes		Particulate Filter Penetration
	Without HEPA Filter	With HEPA Filter	
CCl ₄	85 +	87+	
SO ₂	55 +	57 +	
HCl	80+	87+	
Cl ₂	150+	166+	
ClO ₂	70+	71 +	
H ₂ S	104+	110+	
HCN	40+	41 +	
CH ₂ O	66+	65 +	
NH ₃	46+	48+	
CH ₃ NH ₂	36+	34+	
HF	150+	165 +	
DOP			0.02%
Silica Dust			1.00 mg
Silica Mist			1.75 mg
Lead Fumes			1.05 mg

TABLE 6

BREATHING RESISTANCE OF CARTRIDGES

	Comfo II Without HEPA Filter	Comfo II with HEPA Filter	Adv 100 Without HEPA Filter	Adv 100 with HEPA filter
Breathing Resistance at 42.5 lpm, (mm H ₂ O)	26	40	20	35

Although the present invention has been described in detail in connection with the above examples, it is to be understood that such detail is solely for that purpose and that variations can be made by those skilled in the art without departing from the spirit of the invention except as it may be limited by the following claims.

WHAT IS CLAIMED IS:

1. A filter system for removing toxic agents from gases to be breathed by a user of the filter system, the filter system comprising a container, the container comprising a layer of an activated carbon, the container further comprising a layer of an impregnated activated carbon, the impregnated activated carbon being impregnated with a sulfate compound, a molybdenum compound and at least one compound selected from the group consisting of a copper compound and a zinc compound, the layer of activated carbon comprising at least approximately 20 cc of the activated carbon, and the layer of the impregnated activated carbon comprising at least 80 cc of the impregnated activated carbon.

2. The filter system of Claim 1 wherein the layer of activated carbon comprises at least 26 cc of the activated carbon and the layer of the impregnated activated carbon comprises at least 86 cc of the impregnated activated carbon.

3. The filter system of Claim 1 wherein the activated carbon has a carbon tetrachloride activity of at least 85.

4. The filter system of Claim 3 wherein the activated carbon has a carbon tetrachloride activity of at least 95.

5. The filter system of Claim 1 wherein the impregnated carbon comprises by weight: up to 10% of sulfate compound, up to 10% of molybdenum compound, 0.0 to about 20% of copper compound and 0.0 to about 20% of zinc compound.

6. The filter system of Claim 1 wherein the filter system further comprises a high efficiency particulate filter.

7. The filter system of Claim 1 wherein the layer of activated carbon is positioned downstream from the layer of impregnated activated carbon with respect to the flow of gas through the respirator cartridge upon inhalation by the user thereof.

8. A respirator cartridge for removing toxic agents, including organic vapors, acid gases, formaldehyde, ammonia and methylamine, from gases to be breathed by a user of the respirator cartridge, the respirator cartridge comprising a layer of an activated carbon, the respirator cartridge further comprising a layer of an impregnated activated carbon, the impregnated activated carbon being impregnated with a sulfate compound, a molybdenum compound and at least one compound selected from the group consisting of a copper compound and a zinc compound, the layer of activated carbon comprising at least approximately 20 cc of the activated carbon, and the layer of the impregnated activated carbon comprising at least 80 cc of the impregnated activated carbon.

9. The respirator cartridge of Claim 8 wherein the layer of activated carbon comprises at least 26 cc of the activated carbon and the layer of the impregnated activated carbon comprises at least 86 cc of the impregnated activated carbon.

10. The respirator cartridge of Claim 8 wherein the activated carbon has a carbon tetrachloride activity of at least 85.

11. The respirator cartridge of Claim 10 wherein the activated carbon has a carbon tetrachloride activity of at least 95.

12. The filter system of Claim 8 wherein the impregnated carbon comprises by weight: up to 10% of sulfate compound, up to 10% of molybdenum compound, 0.0 to about 20% of copper compound and 0.0 to about 20% of zinc compound.

13. The respirator cartridge of Claim 8 wherein the respirator cartridge further comprises a high efficiency particulate filter, the respirator cartridge being useful for removing additional toxic agents, including dusts, fumes, radionuclides, lacquers, enamel mists, pesticides and tear gas.

14. The filter system of Claim 8 wherein the layer of activated carbon is positioned downstream from the layer of impregnated activated carbon with respect to the flow of gas through the respirator cartridge upon inhalation by the user thereof.

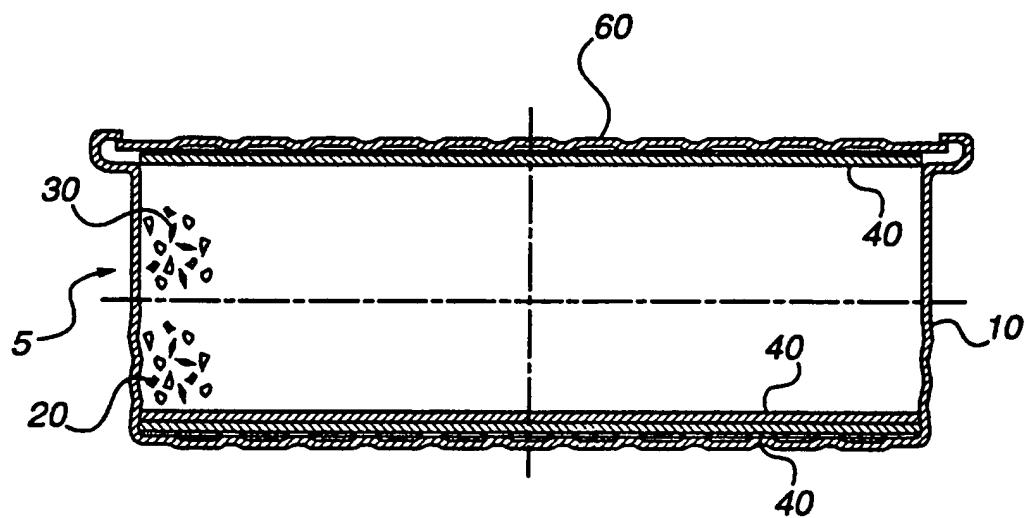


Figure 1

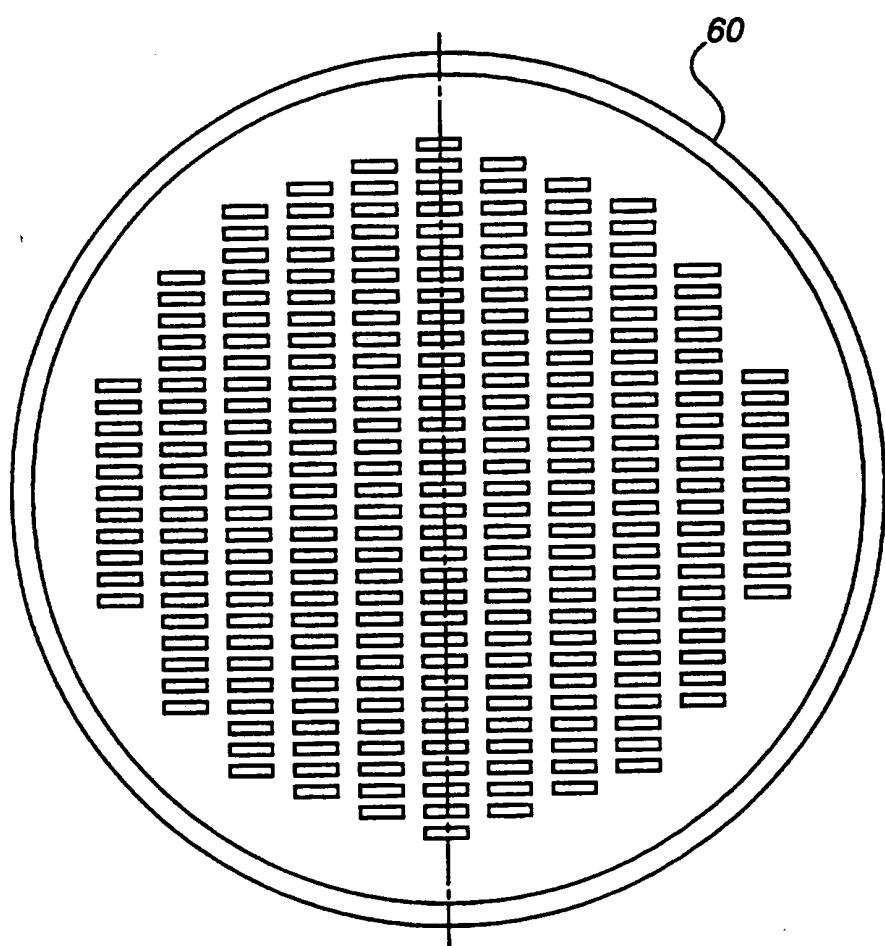


Figure 1A

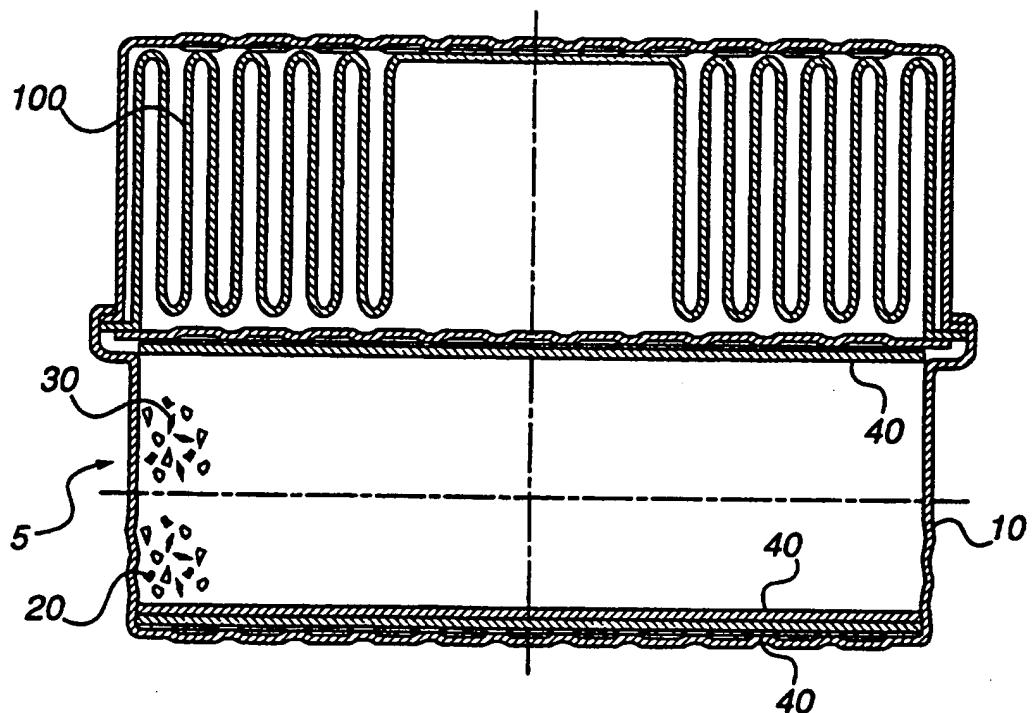


Figure 2

INTERNATIONAL SEARCH REPORT

Int'l Application No
PCT/US 96/00884A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 B01D39/00 A62B23/00 B01D46/24

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 B01D A62B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	EP,A,0 465 371 (INST RECH & SANTE SECURITE) 8 January 1992 see column 2, line 42 - column 3, line 1 see column 4, line 4 - column 6, line 39; claims 1-6; figure 1 ---	1-14
Y	WO,A,93 10896 (CALGON CARBON CORP) 10 June 1993 cited in the application see page 7, line 21 - page 11, line 13; claims 1-12 ---	1-14
A	US,A,5 315 987 (SWANN LINSEY) 31 May 1994 see column 8, line 62 - column 9, line 4; figures 3,4 ---	1-14 -/-

 Further documents are listed in the continuation of box C. Patent family members are listed in annex.

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Date of the actual completion of the international search

Date of mailing of the international search report

18 June 1996

12-07-1996

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INTERNATIONAL SEARCH REPORT

International Application No
PCT/US 96/00884

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP,A,0 405 404 (CALGON CARBON CORP) 2 January 1991 see the whole document ---	1-14
A	EP,A,0 339 487 (BLUECHER HASSO VON ;RUITER ERNEST DE (DE)) 2 November 1989 see the whole document -----	1-14

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/US 96/00884

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